

REMARKS

Entry of the foregoing amendments and favorable reconsideration of the subject application is respectfully requested in view of the following comments.

Claims 1, 2, 5 and 8 are currently pending in the subject application. Claim 1 has been amended herein and new claim 11 has been added. Accordingly, claims 1, 2, 5, 8 and 11 are herewith presented for the examiner's consideration.

The foregoing amendments have been made in response to the examiner's comments in the outstanding Office Action and following review of the specification and claims by Applicants.

The amendment to the specification at the paragraph on page 5, line 20 to page 6, line 18 has been made to correct a typographical error recently discovered in the English translation of the Japanese language specification. Specifically, the minimum value for the molecular weight of the epoxy resin given at the 7th line of the subject paragraph was incorrectly typed as "1000" when it should have been "10,000". This error is readily seen by reference to page 6, line 9 of the Japanese language specification which clearly presents the minimum molecular weight of 10,000 in the corresponding paragraph.

Claim 1 has been amended to specify that the non-crystalline epoxy resin represented by general formula (2) is obtained by reacting a liquid epoxy resin and an aromatic compound represented by general formula (1). In this manner claim 1 is

based on the recitation of Examples 1-3 in which the non-crystalline resin of the present invention is obtained by mixing 189 parts of a liquid epoxy resin and 20, 80 or 109 parts of 3,3',5,5'-tetramethyl-4,4'-dihydroxyldiphenyl methane. In addition, claim 1 has been amended to clarify that the molecular weight distribution is that in the epoxy resin represented by general formula (2), i.e., the non-crystalline epoxy resin of the present invention.

New claim 11, is based on Example 5 of the present specification and is specific to the high-molecular weight epoxy resin of the present invention obtained by reacting the aromatic compound of general formula (1) with an epihalohydrin. Such a high molecular weight resin with a molecular weight distribution comprising an epoxy equivalent of 2,100g/eq or greater is neither disclosed nor suggested by the references of record.

Applicants respectfully submit that the foregoing amendments are properly enterable at this time as they serve to distinguish the present invention over the prior art and/or place the claims in better condition for appeal.

No new matter has been entered by the foregoing amendments.

Rejection of Claims 1 and 5 under 35 U.S.C. § 103(a)

The Office Action rejects claims 1 and 5 under 35 U.S.C. § 103(a) as being unpatentable over Hartmann, U.S. Patent No. 4,153,621 for the reasons stated in the non-final Office Action

of March 14, 2006. That Office Action states:

Hartmann depicts a general structure for the polymerized diglycidyl ether of 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl (cols. 3-4, first general structure) wherein the biphenyl β -hydroxypropylether repeating unit quantified by "n" is zero or an integer utilized in the fabrication of the cured product (col. 6, lines 22-32).

The claimed content of n=0 of at most 60% and an epoxy equivalent weight of at least 250 are not recited.

Hartmann teaches the production of lower molecular weight glycidyl ethers when a ratio of epichlorohydrin to dihydric phenol of higher than 2:1 is employed (col. 3, lines 30-32). "Thus, by decreasing the mol ratio of epichlorohydrin to dihydric phenol from 14 towards two, glycidyl ethers having higher values of n and higher epoxy equivalents can be obtained (col. 3, lines 38-41)".

It would have been obvious to react epichlorohydrin with the 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl at a molar ratio approaching 2:1 in order to mitigate the formation of lower molecular weight glycidyl ethers to within the claimed parameters of at most 60% and to predominantly form higher molecular weight glycidyl ethers with larger epoxy equivalents greater than the claimed minimum of 250 g/eq.

Applicants respectfully traverse the rejection because the *prima facie* case of obviousness has not been established with respect to the presently pending claims 1 and 5.

The Federal Circuit has ruled that a *prima facie* case of obviousness must establish: (1) some suggestion or motivation to modify the references; (2) a reasonable expectation of success; and (3) that the prior art references teach or suggest all claim limitations. Amgen, Inc. v. Chugai Pharm. Co., 18 USPQ2d 1016, 1023 (Feb. Cir. 1991); In re Fine, 5 USPQ2d 1596, 1598 (Fed. Cir. 1988); In re Wilson, 165 USPQ 494, 496 (C.C.P.A. 1970).

A *prima facie* case of obviousness must also include a showing of the reasons why it would be obvious to modify the references to produce the present invention. See Ex parte Clapp, 277 USPQ 972, 973 (Bd. Pat. App. & Inter. 1985). The examiner bears the initial burden to provide some convincing line of reasoning as to why the artisan would have found the claimed invention to have been obvious in light of the teachings. Id. at 974.

Applicants respectfully submit that a *prima facie* case of obviousness has not been established as there is no convincing line of reasoning which would lead one of ordinary skill in the art to apply the teachings of Hartmann to obtain the non-crystalline epoxy resin of the present invention.

Applicants note that there are two methods for producing epoxy resins. The direct method, taught by the Hartmann reference, reacts a divalent phenol and an epihalohydrin, whereas the advanced method of the present application, reacts a divalent phenol and a divalent epoxy resin. With the present amendment of claim 1, claims 1, 2, 5 and 8 are restricted to a non-crystalline epoxy resin which is obtained by the advanced method. In contrast, the reference teaches an epoxy resin obtained by the direct reaction of a divalent phenol, i.e., 3,3',5,5'-tetramethylbiphenol, with an epihalohydrin, i.e., epichlorohydrin, which has a crystalline structure.

In response to Applicants' previous arguments filed August 8, 2006; the examiner contends that nowhere in Hartmann is there

any requirement that the polymerized diglycidyl ether of 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl has to be crystalline. Applicants respectfully submit that the crystalline nature of the epoxy resin obtained by Hartmann is specifically disclosed and can be understood from the presence of melting point data for the resins of the reference. Note that the resin of Example 1 has a melting point of 97°-102°C, those of Examples 2-5 melt at about 100°C, the resin of Example 6 has a melting point of 97°-102°C, Example 7 melts at 107°C, Example 8 melts at 110°-114°C, and Example 9 melts at 104°-105°C.

Furthermore, Applicants respectfully direct the examiner's attention to the following passages from the reference:

In Example 1, col. 9, lines 38-40, "All of the methanol was added when the temperature was at 46°C at which point the product began to crystallize."

In Example 6, col. 10, lines 29-30, "Methanol (1500ml) was added while crystallization took place."

In Example 8, col. 11, lines 59-63, "The product was filtered while hot and purified TMBP-DGE was then crystallized by cooling and storage at 0°C. The recrystallized diglycidyl ether was isolated by filtration and washed with methanol (4x100ml)."

In Example 9, col. 12, lines 37-38, "Crystallized product was filtered and washed with cold cyclohexane/chlorobenzene."

In view of the foregoing, Applicants respectfully submit

that Hartmann is directed to crystalline glycidyl ethers prepared by a reaction of 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl and a halohydrin such as epichlorohydrin and neither teaches nor suggests a non-crystalline epoxy resin as recited in the presently amended claims having a molecular weight distribution comprising content of n=0 component which is 60% or less and an epoxy equivalent which is 250g/eq or over.

In contrast to the teaching of Hartmann, claims 1, 2, 5 and 8 as herein amended are directed to a non-crystalline epoxy resin which is obtained by reacting a liquid epoxy resin and an aromatic compound represented by general formula (1). Indeed, referring to Examples 1-3 of the present application, the liquid resin used as the precursor is that produced in Reference Example 1 from a crystalline diglycidylether resin of 3,3',5,5'-tetramethyl-4,4'-dihydroxyldiphenyl methane, heated to 100°C to fuse the crystalline structure to obtain the liquid form. In the present application, this liquid epoxy resin is then reacted with an aromatic compound represented by general formula (1), which may be an additional quantity of separately prepared 3,3',5,5'-tetramethyl-4,4'-dihydroxyldiphenyl methane, in the presence of nitrogen and butyltriphenylphosphoniumbromide, to obtain the non-crystalline solid or semi-solid resin. In that regard, Applicants respectfully submit that the present invention presents a composition that is different in structure and properties from that of Hartmann and which is produced by a method which is, essentially, a further process beyond that disclosed by Hartmann

since the liquid epoxy resin which is the precursor in the present invention is equivalent to the diglycidyl ether of 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl product of Hartmann. Thus, since Hartmann neither discloses nor suggests such a further process treating the diglycidyl ether of 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl product, Hartmann neither discloses nor suggests a non-crystalline epoxy resin produced by such further process and having the claimed properties.

At best, Hartmann discloses using the non-crystallized, methanol soluble residual fraction from the preparation of the crystalline resin of Example 1 and having an epoxy equivalent of 272 in Example 5 where it is mixed with a curing agent identified as either methylene dianiline or m-phenylene diamine. There is no disclosure to suggest mixing that residual fraction or the actual product of Example 1 with an aromatic compound of general formula 1. The fact that this residual fraction has an epoxy equivalent of 272 is not relevant since it was obtained by a process dissimilar to that recited in the present claims.

In the absence of some suggestion that the product of Hartmann undergo further processing with an additional aromatic compound corresponding to the present claims and resulting in a non-crystalline epoxy resin having the claimed molecular weight distribution, a prima facie showing of obviousness of claims 1 and 5 has not been made. Accordingly, Applicants respectfully submit that the examiner's rejection is without support and should be withdrawn.

Rejection of Claims 1, 2, 5 and 8 under 35 U.S.C. § 103(a)

The Office Action rejects claims 1, 2, 5 and 8 under 35 U.S.C. § 103(a) as being unpatentable over Kawano, et al., U.S. Patent 7,063,914 (U.S. Pub. 2003/0175571) in view of Hartmann, U.S. Patent No. 4,153,621 for the reasons stated in the non-final Office Action of March 14, 2006. That Office Action states:

Kawano, et al. (page 1, paragraphs 6 and 7, general formula 1) exhibits an optionally C₁₋₆ hydrocarbon-substituted bisphenol F diglycidyl ether wherein the β -hydroxypropylether diphenyl methane repeating unit is as high as 15 used in a cured product (page 3, paragraph 30).

The claimed content of n=0 of at most 60% and an epoxy equivalent weight of at least 250 are not recited.

It would have been obvious to prepare the polymerized diglycidyl ether of bisphenol F with a molar ratio of near 2:1 as espoused in Hartmann in order to reduce the yield of lower molecular weight glycidyl ethers to within the claimed range of at most 60% and to produce a predominance of higher molecular weight glycidyl ethers with larger epoxy equivalents exceeding the claimed minimum of 205g/eq."

Applicants respectfully traverse the rejection because the *prima facie* case of obviousness has not been established with respect to the presently pending claims 1, 2, 5 and 8.

The Federal Circuit has ruled that a *prima facie* case of obviousness must establish: (1) some suggestion or motivation to modify the references; (2) a reasonable expectation of success; and (3) that the prior art references teach or suggest all claim limitations. Amgen, Inc. v. Chugai Pharm. Co., 18 USPQ2d 1016, 1023 (Feb. Cir. 1991); In re Fine, 5 USPQ2d 1596, 1598 (Fed. Cir.

1988); In re Wilson, 165 USPQ 494, 496 (C.C.P.A. 1970).

A *prima facie* case of obviousness must also include a showing of the reasons why it would be obvious to modify the references to produce the present invention. See Ex parte Clapp, 277 USPQ 972, 973 (Bd. Pat. App. & Inter. 1985). The examiner bears the initial burden to provide some convincing line of reasoning as to why the artisan would have found the claimed invention to have been obvious in light of the teachings. Id. at 974.

Applicants respectfully submit that a *prima facie* case of obviousness has not been established as there is no convincing line of reasoning which would lead one of ordinary skill in the art to apply the teachings of Hartmann to obtain the non-crystalline epoxy resin of the present invention.

As with Hartmann, there is no disclosure in Kawano, et al., which suggests formation of the non-crystalline epoxy resin of the present invention in the manner recited in claim 1 where a liquid epoxy resin is reacted with an aromatic compound represented by general formula (1) so as to have the recited characteristics. At best Kawano, et al., follows the direct method of Hartmann in which bisphenol F or a substituted bisphenol F is epoxidized with epichlorohydrin.

Applicants again point out that the C₁₋₆ hydrocarbon substituted bisphenol F diglycidyl ether of Kawano, et al., is a crystalline epoxy resin and is different from the non-crystalline epoxy resin of the present invention. Indeed, the substituted

bisphenol F resin used in Example 2 of the reference is identified as a tetramethylbisphenol F type epoxy resin with the commodity name of YSLV-80XY available from Nippon Steel Chemical with a melting point of 75-80°C. This is substantially the same as the diglycidylether resin of 3,3',5,5'-tetramethyl-4,4'-dihydroxyldiphenyl methane (YSLY-80XY) used in Reference Example 1 of the present invention (page 10, line 25 to page 11, line 3) which is a product of Shin-Nittetsu Chemicals, Co., Ltd., and which is the precursor liquid epoxy resin used in Examples 1-3 of the present invention to produce the non-crystalline epoxy resin recited in the claims.

As with Hartmann, Kawano, et al., neither discloses nor suggests reacting such a liquid epoxy resin with an aromatic compound represented by general formula (1) to produce the non-crystalline epoxy resin of the present invention.

Since the tetramethyl bisphenol F epoxy resin of the Reference Example 1 of the present application is substantially the same as that disclosed by Kawano, et al., Applicants respectfully submit that the reference merely discloses the resin used by Applicants as a reference against which the non-crystalline epoxy resin of the present application is compared. The reference neither discloses nor suggests the non-crystalline epoxy resin of the present invention prepared as recited in claim 1 and having a content of n=0 component of 60% or less and an epoxy equivalent of 250 g/eq or more. For the reasons given previously herein, the addition of the Hartmann reference does

not cure the deficiencies of the Kawano, et al., reference since Hartmann fails to disclose or teach a non-crystalline epoxy resin represented by general formula (2) as recited in the present claims having the content of n=0 component of 60% or less and an epoxy equivalent of 250 g/eq or more.

Applicants point out that, in both instances, the examiner has acknowledged that neither reference recites the claimed content of n=0 of at most 60% and an epoxy equivalent weight of at least 250 in the product compositions of the references. For the examiner to suggest that such properties would be obvious when there is no support therefore in the prior art of record borders on impermissible hindsight, particularly where, as noted herein, the non-crystalline epoxy resin of the present claims is a different composition from that presented by the references and is prepared in a process which is essentially a further step applied to the epoxy resins of the references but neither disclosed nor suggested therein.

In view of the foregoing, Applicants respectfully submit that there is no motivation in either Hartmann or Kawano, et al. to modify the teachings therein to achieve the non-crystalline epoxy resin of the present invention.

Accordingly, Applicants respectfully submit that the rejection of claims 1, 2, 5 and 8 over Kawano, et al., in view of Hartmann is without support and should be withdrawn.

With regard to the examiner's additional comments in the present office action, Applicants have addressed the issue of the

crystallinity of the Hartmann resin and respectfully direct the examiner's attention to the passages of Hartmann cited herein. As for the examiner's contention that Hartmann contemplates diglycidyl ethers with higher values of n and higher epoxy equivalents and that the reference displays a "lack of aversion to species of high epoxy equivalent", it is noted that the examiner has already admitted that the reference does not recite the presently claimed content of n=0 of at most 60% and an epoxy equivalent of at least 250. Furthermore, as pointed out in Applicants' response of August 8, 2006, the desirable embodiment of Hartmann is 3,3',5,5'-tetraalkyl-4,4'-dihydroxybiphenyl (TMBP-DGE) characterized by having a content of n=0 component of 88% or more and epoxy equivalents of less than 200g/eq (Applicants' response 8/8/06, pg. 14-16). Accordingly, Applicants respectfully submit that the reference's teaching of epoxy resins having a content of n=0 component of greater than the claimed 60% and epoxy equivalent of less than the claimed minimum of 250g/eq outweighs any supposition on the part of the examiner.

The examiner notes that example 1 (col. 9, line 68) of Hartmann produces a diglycidyl ether of 3,3',5,5'-tetramethylbiphenol having an epoxide equivalent of 272. Applicants respectfully point out that this is, in fact, a liquid epoxy residue from the separation of the actual crystalline TMBP-DGE product of Example 1 by filtration and, as previously pointed out herein, Hartmann fails to disclose or suggest a further processing of this liquid fraction with an aromatic compound of

general formula 1 as recited in the present claims. Furthermore, the liquid fraction is only obtained upon separation from the total epoxy resin formed, accordingly, only 18.7% of the resin obtained in Example 1 of Hartmann exhibits an epoxy equivalent of 272. Since the component of the crystal form (n=0) is 81.3%, the total epoxy equivalent of the resin produced by Example 1 is 201.39g/eq ($100 / (81.3/190 + 18.7/272) = 201.39\text{g/eq}$) which is below the recited minimum of the present application.

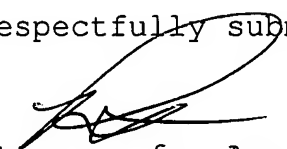
With regard to the examiner's comments concerning the criterion for distinguishing the flexibility of the diglycidyl ether of tetramethylbisphenol F (YSLV-80XY represented by Comparative Example 3) from that of the tetramethylbisphenol F-advanced diglycidyl ether of bisphenol F of Example 8 in the present application, Applicants point out that this flexibility is evaluated by the JIS-G5528 5.4.4 standard which relates to epoxy-powder coating for interiors of ductile iron pipes and fittings. The 5.4.4 standard is determined by the Erichsen Test prescribed in JIS-Z2247 as the Erichsen Test Method and establishes the distance to push in a punch as 3mm. Accordingly, the difference between a result of 3.1 for Example 8 as compared to 3.0 for comparative example 3 is significant since it indicates whether or not the flexibility of the coating passes or fails the JIS-G5528 standard. For the examiner's convenience, copies of JIS-G5528 and JIS-Z2247 are submitted herewith.

With regard to Reference Example 1 (pages 10-11), Applicants point out that the addition of 1g of fine crystal grain of

diglycidylether resin of 3,3',5,5'-tetramethyl-4,4'-
dihydroxyldiphenylmethane to the fused crystalline liquid epoxy
resin is a reconfirmation of the crystallinity of the YSLV-80XY.
The small amount of fine crystal grain of the resin added does
not change the n=0 content of 90%. In contrast, the addition of
the higher quantities of 3,3',5,5'-tetramethyl-4,4'-
dihydroxyldiphenyl methane to the liquid epoxy resin in Examples
1-3 result in the final non-crystalline resin having a content of
the n=0 component of 60% or less and epoxy equivalent of 250g/eq
or over.

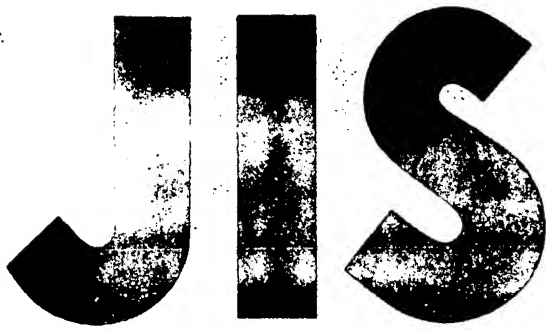
In view of the foregoing, Applicants respectfully submit
that the non-crystalline epoxy resin of the present claims is
neither disclosed nor suggested by the prior art nor are the
improved results obtained with the epoxy of the present
invention. Applicants therefore respectfully submit that the
examiner's rejections should be withdrawn and that the present
application is now in condition for allowance. An early notice
of allowance is earnestly solicited.

Respectfully submitted,



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JAPANESE INDUSTRIAL STANDARD

Epoxy-Powder Coating for Interior of Ductile Iron Pipes and Fittings

JIS G 5528—1984

JIS G 5528:1984 has been revised under date of February 20, 2006. The revised items are included in Amendment 1.

Translated and Published

by

Japanese Standards Association

Translation without guarantee
In the event of any doubt arising, the original
standard in Japanese is to be evidence

JAPANESE INDUSTRIAL STANDARD

J I S

Epoxy-Powder Coating for Interior of
Ductile Iron Pipes and Fittings

G 5528-1984

1. Scope

This Japanese Industrial Standard specifies the epoxy-powder paint films, hereinafter referred to as the "films", formed on the inner surface of ductile iron pipes⁽¹⁾, hereinafter referred to as the "pipes", and their coating methods.

Note (¹) Ductile iron pipes herein mean ductile iron pipes and ductile iron fittings.

2. Coating

The coating shall not contain injurious components to use, being not dissolved with water after curing, exerting no bad influence upon the quality of water and shall have the composition of (1) and the quality of (2).

- (1) Composition The composition of coating shall be the thermosetting powdery coating using epoxy resin, hardener and pigment as main raw materials.
- (2) Quality The quality of coating shall be as given in Table 1.

Applicable Standards:

- JIS G 5526-Ductile Iron Pipes
- JIS G 5527-Ductile Iron Fittings
- JIS K 5101-Testing Methods for Pigments
- JIS K 5400-Testing Methods for Organic Coatings
- JIS Z 2247-Method of Erichsen Cupping Test

Table 1. Quality of Coating

	Quality specification																
Specific gravity of film	When the test of 5.4.1 is carried out, the specific gravity shall be 1.8 max.																
Adhesion of film	When the test of 5.4.2 is carried out, the evaluation merit marks shall be 8 min.																
Impact resistance of film	When the test of 5.4.3 is carried out, cracking and peeling shall not be generated due to the deformation by impact.																
Flexibility of film	When the test of 5.4.4 is carried out, cracks shall not be generated.																
Scratch resistance of film	When the test of 5.4.5 is carried out, there shall be no abnormality.																
Corrosion resistance of film	When the test of 5.4.6 is carried out, there shall not be any rust, blistering, cracking or the like.																
Temperature resistance repeatability of film	When the test of 5.4.7 is carried out, crinkling, cracking, blistering, peeling or the like shall not be generated and discoloration shall not be great.																
Elution of film ⁽²⁾	<p>When the test of 5.4.8 is carried out, its results shall be as follows:</p> <table> <tr> <td>Turbidity</td><td>0.5 degree max.</td></tr> <tr> <td>Chromaticity</td><td>1 degree max.</td></tr> <tr> <td>Consumption of potassium permanganate</td><td>2 mg/l max.</td></tr> <tr> <td>Loss of residual chlorine</td><td>0.7 ppm max.</td></tr> <tr> <td>Phenol groups</td><td>0.005 mg/l max.</td></tr> <tr> <td>Amine</td><td>No detection</td></tr> <tr> <td>Cyan</td><td>No detection</td></tr> <tr> <td>Odour and taste</td><td>No abnormality</td></tr> </table>	Turbidity	0.5 degree max.	Chromaticity	1 degree max.	Consumption of potassium permanganate	2 mg/l max.	Loss of residual chlorine	0.7 ppm max.	Phenol groups	0.005 mg/l max.	Amine	No detection	Cyan	No detection	Odour and taste	No abnormality
Turbidity	0.5 degree max.																
Chromaticity	1 degree max.																
Consumption of potassium permanganate	2 mg/l max.																
Loss of residual chlorine	0.7 ppm max.																
Phenol groups	0.005 mg/l max.																
Amine	No detection																
Cyan	No detection																
Odour and taste	No abnormality																

Note (2) When used for water service, the elution of film is tested.

3. Quality of Film of Product

The quality of film of product shall be as given in Table 2.

Table 2. Quality of Film of Product

Quality Item	Quality specification
Appearance of film	When the test of 6.2 is carried out, there shall not be any mixing of foreign matters, coating unevenness, coating lack or the like and its surface shall be smooth and uniform. Further, the pinholes shall be free from defects likely to generate sparks.
Adhesive property of film	When the test of 6.3 is carried out, there shall be no flaking.
Curing degree of film	When the test of 6.4 is carried out, chips and peeling shall not be generated.
Thickness of film	The film thickness of product shall be 0.3 mm min. at the part B given in Attached Fig. However, the dimension of D_1 of JIS G 5526 and JIS G 5527 after coating shall be within its permissible range.
Scratch resistance of film	When the test of 6.6 is carried out, it shall conform to the requirements specified in Table 1.

4. Coating Method

4.1 Pretreatment of Coating Surface The pretreatment of coating surface shall be as follows:

- (1) Coating rats, rust, other adhesives injurious for coating or the like shall be removed by using a grinder, a sander or the like and finished to be as flat and smooth as possible.
- (2) The pretreated cast iron surface shall be protected not so as to be rusted again or attached with dust, oil content or the like.

4.2 Coating Preparation The coating shall be used within an available period designated by the manufacturer of coating.

Further, when recovered coatings are used, they can be used by compounding them with 50 % or under of new coating after removing foreign matters using a sieve of 150 to 220 μm .

4.3 Coating The coating shall be carried out as follows:

- (1) For coating, a preheated pipe is sprayed with coating using a suitable powder coating equipment and film is formed. The preheat temperature shall be as specified by the manufacturer of coating.

Further, for the pipe finished with coating, the film shall be sufficiently cured.

- (2) The coating shall be carried out so that it is free from defects such as mixing of foreign matters, coating unevenness, pinholes, coating lack or the like, and the smooth and uniform film on surface can be obtained.

4.4 Range of Coating The range of coating shall be as given in Attached Fig.

4.5 Repair of Coating The repair of coating may be carried out by using ordinary temperature thermosetting epoxy resin series coating by coating traders when light defects not conforming to the requirements specified in 3. on the results of the test of 6. are approved by the purchaser.

Further, in the case of straight pipe, after polishing the coating surface by using a grinder, a sander or the like, it may be repaired by recoating it according to 4.3.

5. Test of Coating

5.1 General Conditions for Test The general conditions for test shall be in accordance with 3. of JIS K 5400.

Further, the test shall be carried out by the manufacturer of coating and its test results sheet shall be presented to coating traders.

Furthermore, when considered to be necessary by purchaser, the purchaser may require its test results sheet to be presented as well as attending the test.

5.2 Sampling Method of Coating The coating shall be sampled in accordance with 2. of JIS K 5400 for every manufacturing lot.

5.3 Preparation of Coating Test Pieces

5.3.1 Materials, Dimensions and Number of Test Pieces for Each Test Item
The materials, dimensions and number of test pieces shall be as given in Table 3.

Table 3. Materials, Dimensions and Number of Test Pieces for Each Test Item

Test item	Materials of test piece	Dimensions of test piece mm	Number	Lot size
Cross-cut test	Steel plate	150 x 70 x 2.0	3	Production lot
Impact deformation test			3	
Pencil scratch test			1	
Salt spray test			3	The lot of the same coating manufactured in 6 months
Low and high temperatures repeat test			2	
Erichsen cupping test	Pipe	90 x 90 x 1.2	1	Production lot
Dissolution test		Nominal diameter 75 x 500	1	The lot of the same coating manufactured in 6 months

5.3.2 Preparation of Test Piece The preparation of test piece shall be carried out as follows:

- (1) In the Case of Steel Plate It is coated with film of 0.2 mm in thickness according to 4.3 by using the steel plate specified in Table 3 and cooled to room temperature.
- (2) In the Case of Pipe The inner surface of pipe is coated with film of 0.3 mm in thickness according to 4.3 by using the pipe specified in Table 3 and cooled to room temperature.

5.4 Test Methods

5.4.1 Test for Specific Gravity of Film The test for specific gravity of film shall be carried out in accordance with 17. of JIS K 5101.

5.4.2 Cross-cut Test The cross-cut test shall be carried out in accordance with 6.15 of JIS K 5400.

5.4.3 Impact Deformation Test The impact deformation test shall be carried out in accordance with Method B of 6.13 of JIS K 5400. However, the falling height shall be 50 cm.

5.4.4 Erichsen Cupping Test The Erichsen cupping test shall be carried out in accordance with Method A of JIS Z 2247. However, the depth where the punch is pushed in, shall be 3 mm.

5.4.5 Pencil Scratch Test The pencil scratch test shall be carried out in accordance with 6.14 of JIS K 5400. However, the pencil of H in hardness shall be used.

5.4.6 Salt Spray Test The salt spray test shall be carried out in accordance with 7.8 of JIS K 5400 and the absence of rust, blistering, cracking or the like shall be affirmed after 500 h. However, the test piece shall be free from scratches.

5.4.7 Low and High Temperatures Repeat Test For the low and high temperatures repeat test, the state of film regarding two test pieces shall be examined after carrying out the following operation:

At first, after keeping the test piece in a thermostat kept at $20 \pm 1^\circ\text{C}$ for 2 h, it is kept in a thermostat at $-30 \pm 1^\circ\text{C}$ for 2 h. Then, after keeping it in the thermostat kept at $20 \pm 1^\circ\text{C}$ for 1 h, it is kept in a thermostat kept at $70 \pm 1^\circ\text{C}$ for 2 h and further kept in the thermostat kept at $20 \pm 1^\circ\text{C}$ for 17 h. Those operation are made one cycle and the test is carried out by repeating 4 cycles.

5.4.8 Dissolution Test The bottom is attached to the one end of test piece by a rubber stopper of good quality covered with polyvinylidene chloride film⁽³⁾ and it is washed in running water (service water) for 1 h. Thereafter, it is emptied once, washed with specimen water, then filled with specimen water, its upper part being covered with polyvinylidene chloride film and stood it still in a dark place at ordinary temperature for 24 h. Thereafter, that water is made the sample water.

Further, a beaker of 2 l is filled with specimen water, its upper part being covered with polyvinylidene chloride film, it is stood still at the same position as that of sample water for 24 h and it is made the blank test water. The preparation method and test method of specimen water shall be in accordance with Appendix.

Further, the test temperature shall be ordinary temperature and the numerical values given in Table 1 or the like shall be obtained from the difference from that of blank test.

Note (3) The polyvinylidene chloride film to be used shall be preliminarily washed with nitric acid (5 %) and then left washed with water as it is.

6. Test for Film of Product

6.1 General Conditions of Test The general conditions of test shall be as follows:

- (1) The range of test of film shall be as given in Attached Figure.
- (2) The test shall be carried out by coating trader and purchaser and the number of tests shall be total of tests except curing test.

6.2 Appearance Test The appearance test shall be carried out according to the following method:

- (1) The test of mixing of foreign matters, coating unevenness and coating lack shall be carried out by visual observation.

- (2) Pinhole test shall be carried out with a holiday detector by applying the voltage of 1000 V thereto.

6.3 Adhesive Property Test The adhesive property test shall be carried out by slightly striking the coating surface with a test hammer.

6.4 Curing Test For the curing test, in the case of pipe of 150 mm max. in inner surface of receiving opening part or nominal diameter, the film coated on the position of outer surface especially specified is cut with 2 lines of scratch (cross-cut) of 25 mm in length reaching basis material intersecting by 30° using a cutter knife specified in (2.1) of 6.15 of JIS K 5400 or the like and the presence of lacks and peelings of film shall be examined.

The number of tests in that case shall be one per each group by making 10 pieces and its fraction of the pipe having the same nominal diameter of coating pipe per day one group.

Further, after test, the cross-cut part shall be repaired by using the same coating as that for outer surface coating.

6.5 Measurement of Thickness of Film The measurement of thickness of film shall be carried out as follows:

- (1) The thickness of film shall be measured using an electromagnetic micrometer or other suitable measuring instruments. For its measuring positions, optional 2 positions are determined in the longitudinal direction and optional 4 points on the circumference of those positions shall be selected.
- (2) The dimension of receiving opening inner diameter shall be measured by using minimum limit gauge or the like.

6.6 Pencil Scratch Test The pencil scratch test shall be carried out according to the method of 5.4.5, as appropriate.

7. Retest

The retest shall be carried out in accordance with the requirements specified in 6. in the case where the repair of coating of 4.5 is carried out.

8. Inspection

8.1 Inspection of Coating The inspection of coating shall be carried out in accordance with 5. regarding specific gravity of film, adhesion of film, impact resistance of film, flexibility of film, scratch resistance of film, corrosion resistance of film, temperature resistance repeatability of film and elution of film, and the results shall comply with the specifications of 2.

8.2 Inspection on Film of Product The inspection on film of product shall be carried out in accordance with 6. and 7. regarding appearance of film, adhesive property of film, curing degree of film, thickness of film and scratch resistance of film and shall conform to the requirements specified in 3.

9. Marking

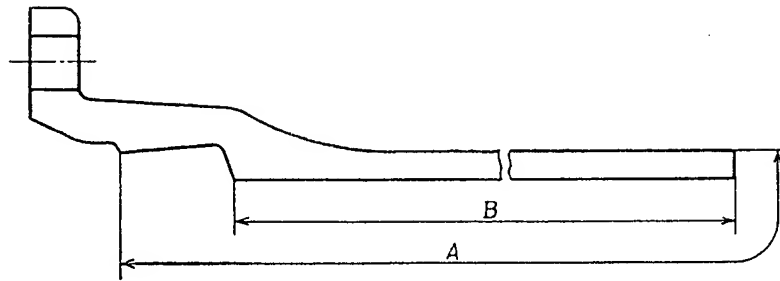
The product shall be marked on the legible position of the inner surface on the receiving opening side of product by an indelible method with the following information:

- (1) Year and month of coating or its mark
- (2) Coating trader's name or mark

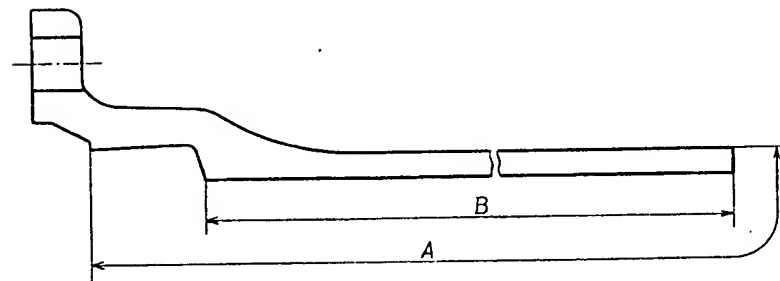
10. Protection of Coating Surface

The product accepted by inspection shall be suitably capped on the receiving opening and insertion opening parts to protect the coating surface.

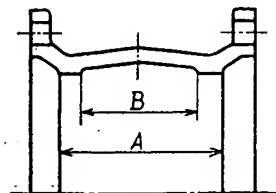
Attached Figure. Range of Coating and of Inspection of Coating
In the Case of Type K



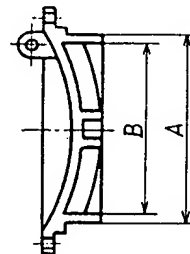
In the Case of Type A



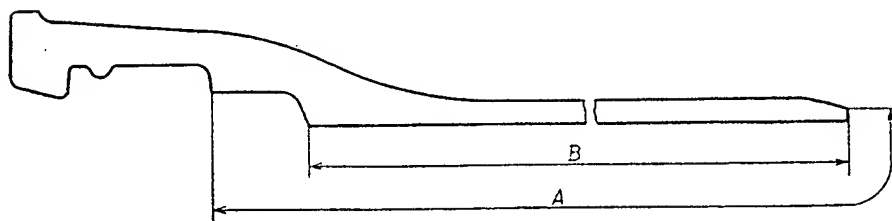
Coupling Ring



Stopper

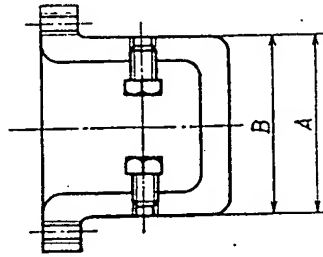


In the Case of Type T

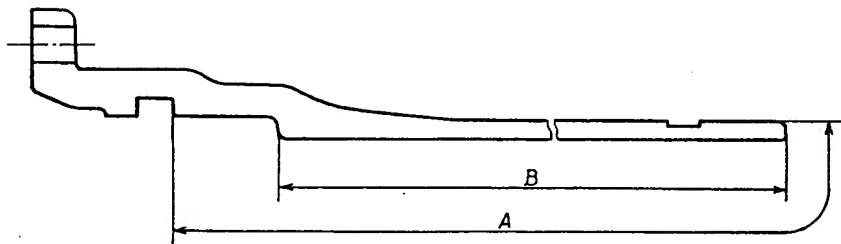


Attached Figure (Continued)

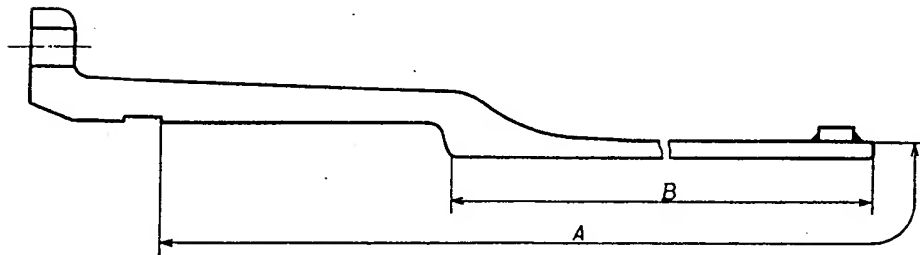
Stopper



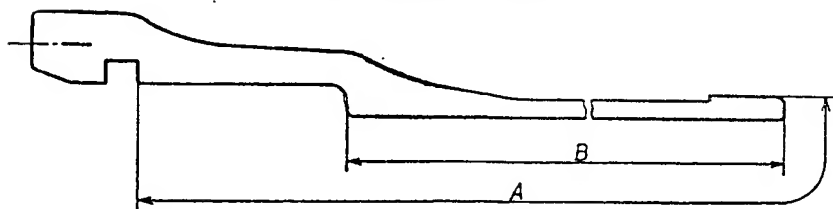
In the Case of Type KF



In the Case of Type SII

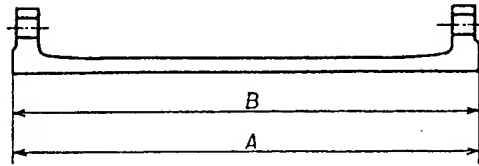


In the Case of Type S



Attached Figure (Continued)

In the Case of Flange Type



- Remarks 1. The range of coating shall be part *A*.
2. The range for inspection of coating shall be part *B*.
3. The part except the range of *B* within the range of *A* shall generally be further processed with the same coating as that of outer surface.

Appendix. Methods for Dissolution Test of Epoxy-powder Coating for Interior of Ductile Iron Pipes and Fittings

1. Preparation Methods of Specimen Water

1.1 Reagents

1.1.1 Lime Water Take about 100 parts of purified water into a suitable vessel, add 2 parts of slaked lime, stopper it, sufficiently shake it and stand it still for 24 h. Thereafter, filter the supernatant into the vessel preliminarily put with about 100 parts of purified water using a filter paper (Class 5 C), store it with stoppering and allow it to be the lime water. If films or precipitates are generated, filter them.

To determine the calcium in the lime water, take about 100 ml of purified water into an Erlenmeyer flask (300 ml) and add 1 ml of about 0.1 % magnesium chloride (MgCl_2) solution, 2 ml of ammonia buffer solution and 5 to 6 drops of EBT indicator. Thereafter, drip 0.01 M-EDTA solution until the solution turns blue (take cares about excess dripping).

Then, accurately add 1 ml of lime water to the solution, titrate the wine red coloured solution with 0.01 M-EDTA solution, obtain the amount (a , ml in unit) of EDTA solution required therefore and calculate the calcium concentration (ppm) of lime water from the following formula:

$$\text{Calcium in lime water (Ca ppm)} = a \times \frac{40}{100} \times 1000$$

1.1.2 Purified Water It is the distilled water or the desalted water by passing it through ion exchange resin layer, of which conductivity is $3 \mu\Omega/\text{cm}$ max.

1.1.3 Ammonia Buffer Solution Take 67.5 g of ammonium chloride (NH_4Cl) into a beaker and dissolve it with about 300 ml of purified water. Thereafter, add 570 ml of aqueous ammonia (28 %) and dilute it to 1 l further by using purified water.

1.1.4 EBT Indicator Dissolve 0.5 g of Eriochrome Black T and 4.5 g of hydroxylamine hydrochloride with 100 ml of ethyl alcohol (95 %) and store it in a brown bottle (valid for about 1 month).

1.1.5 0.01 M-EDTA Solution Take 4 g of disodium ethylenediaminetetraacetate [$(\text{CH}_2\text{COO})_2\text{N}\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{N}(\text{CH}_2\text{COO})_2\text{H}_2\text{Na}_2\cdot 2\text{H}_2\text{O}$] and dilute it to 1 l by dissolving it with purified water.

To determine the factor of this solution, accurately take 25 ml of 0.01 M zinc chloride solution into an Erlenmeyer flask (300 ml) and add about 75 ml of purified water, 2 ml of ammonia buffer solution and 5 to 6 drops of EBT indicator. Thereafter, titrate it using above-mentioned EDTA solution until its wine red turns blue, obtain the amount of EDTA solution (a , ml in unit) required therefore and calculate the factor from the following formula:

$$\text{Factor (F)} = \frac{25}{a}$$

Then, take 1000/F ml of above-mentioned EDTA solution into a measuring flask (1 l) and dilute whole quantity thereof to 1 l by adding purified water.

1.1.6 0.01 M Zinc Chloride Solution Take 0.654 g of the standard reagent zinc (Zn) into a beaker, which is preliminarily washed with hydrochloric acid (1 + 3), purified water and acetone in this order and immediately thereafter dried in a calcium chloride desiccator or sulfuric acid desiccator for 24 h min., add about 20 ml of purified water and 3 ml of hydrochloric acid (35 %) and dissolve it by heating on water bath:

After cooling, transfer it into a measuring flask (1 l), sufficiently wash the beaker with purified water, put the washings into the measuring flask and make the whole amount thereof 1 l by adding purified water.

1.2 Preparation of Specimen Water Add lime water to purified water so that the amount of calcium becomes 12 ppm (30 ppm in hardness), allow it to be 8.0 to 7.5 in pH by passing carbonic acid gas through it, add chlorine thereto, stand it still for 12 to 24 h and allow it to contain free residual chlorine of 1.0 to 1.2 ppm.

Then, take a portion (1/5 to 1/10) of this solution into another vessel, prepare the solution of low pH by passing carbonic acid gas, regulate it so that its pH becomes 7.0 ± 0.2 little by little adding this to the original solution and allow this solution to be the specimen water.

2. Test Methods

2.1 Turbidity The turbidity means the turbidity of water and the turbidity where 1 l water contains 1 mg kaolin shall be taken as 1 degree.

2.2 Reagents and Device The reagents and device shall be as follows:

- (1) **Kaolin for Turbidity** Take about 10 g of kaolin on the market into a beaker of 500 ml, add 300 ml of purified water and 0.2 g of sodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10 \text{H}_2\text{O}$) (a) and mix with violently stirring them for 3 min using a stirrer or the like. Transfer it to a measuring cylinder with ground stopper (1 l), sufficiently wash the beaker with water, add the washings to the measuring cylinder, make its total amount 1 l by adding purified water, sufficiently mix it by shaking for 1 min and disperse it.

After standing it still at ordinary temperature for 1 h, discard the liquid of 250 ml from the surface by using a syphon and sample 500 ml (b) below that into an evaporating dish of known amount. Evaporate the sampled solution on water bath to dryness, further dry it at 105 to 110°C for about 3 h and leave it cool in a desiccator. Thereafter, obtain the mass (c) of kaolin for turbidity by measuring. Then, grind it in an agate mortar and store it in a wide-mouthed bottle.

The mass (d) of pure kaolin thus obtained, is

$$d = c - \left(a \times \frac{b}{1000} \times \frac{141.959 \times 2}{446.057} \right)$$

Therefore, to weigh out 1 g of pure kaolin, c/d g of kaolin for turbidity may be taken.

- (2) Turbidity Standard Stock Solution Take c/d g of kaolin for turbidity into a measuring flask (1 l), add 10 ml of formalin thereto, make its total amount 1 l using purified water and allow that to be the stock solution.

Accurately take 100 ml of that solution into another measuring flask (1 l) while sufficiently mixing this stock solution with shaking and dilute it to 1 l using purified water.

Allow 1 ml of this solution to contain 0.1 mg kaolin.

- (3) Test Tube It is the flat bottom non-coloured test tube with ground stopper of about 37 cm in total length of which the bottom surface is polished, attached with a marked line of 100 ml at a height of 30 cm from bottom.

2.3 Test Operation Respectively take 100 ml of sample water and blank test water into test tubes.

Separately take a suitable amount (0.5 to 10 ml) of turbidity standard stock solution into several test tubes having the same shape while sufficiently mixing with shaking the turbidity standard stock solution, dilute them to 100 ml by adding purified water, stopper it, mix it with gently shaking and allow that to be the standard solution.

Then, place them on a black paper, compare the turbidity of sample water and blank test water with that of standard solution by seeing through them from upward, obtain the amount (ml) of turbidity standard solution corresponding thereto and calculate the turbidity of sample water from the following formula:

$$\text{Turbidity (degree)} = \left(a \times \frac{1000}{S} \times 0.1\right) - \left(b \times \frac{1000}{B} \times 0.1\right)$$

where a : turbidity standard stock solution corresponding to the turbidity of sample water (ml)

b : turbidity standard stock solution corresponding to the turbidity of blank test water (ml)

S : sample water (ml)

B : blank test water (ml)

3. Chromaticity

The chromaticity means the degree of colour of water and the colour appearing where 1 ml of chromaticity standard stock solution is diluted to 1 l by water, shall be taken as 1 degree.

(1) Reagent and Device

- (1.1) Chromaticity Standard Stock Solution Take 2.49 g of potassium chloroplatinate (K_2PtCl_6) (containing 1 g of Pt), 2.02 g crystalline cobalt chloride ($CoCl_2 \cdot 6 H_2O$) (containing 0.5 g of Co) and 200 ml hydrochloric acid (35 %) into a measuring flask (1 l), dissolve them with purified water and make its whole amount 1 l. Allow 1 ml this solution to contain 1 mg of platinum (Pt).

(1.0) Test Tube It is in accordance with 2.2 (3).

- (2) Test Operation Respectively take 100 ml of sample water and blank test water into test tubes. Separately, take a suitable amount (0.1 to 1.5 ml) of chromaticity standard stock solution into several same type test tubes, dilute it to 100 ml by adding purified water, stopper it, mix it with quietly shaking and allow that to be the standard solution.

Then, lay those on white paper, compare the hues of test water and blank test water with that of standard solution by seeing through those from the upward, obtain the number of ml of chromaticity standard stock solution and calculate the chromaticity of sample water from the following formula:

$$\text{Chromaticity (degree)} = \left(a \times \frac{1000}{S} \right) - \left(b \times \frac{1000}{B} \right)$$

where a : chromaticity standard stock solution corresponding to the hue of sample water (ml)

b : chromaticity standard stock solution corresponding to the hue of blank test water (ml)

S : sample water (ml)

B : blank test water (ml)

4. Consumption of Potassium Permanganate

4.1 Reagents and Devices The reagents and devices shall be as follows:

- (1) Dilute Sulfuric Acid After slowly adding 1 volume part of sulfuric acid (95 %) into two volume parts of purified water while mixing them with stirring, drop potassium permanganate solution (0.5 %) while warming it until faint red does not disappear and remains.
- (2) 0.01 N Sodium Oxalate Solution Take 0.670 g of standard reagent sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) which is dried at 150 to 200°C for 1 to 1.5 h and left cool in a desiccator, into a measuring flask (1 l), dissolve it with purified water to make its total amount 1 l and store it in a brown bottle (that elapsed about 1 month min. shall not be used).

One ml of this solution corresponds to 0.316 mg of potassium permanganate.

- (3) 0.01 N Potassium Permanganate Solution Take 0.32 to 0.34 g of potassium permanganate (KMnO_4) into a beaker (2 l), add 1050 ml of purified water and gently boil it for 1 to 2 h. Thereafter, leave it as it is in a dark place for one night, then filter the supernatant with a glass filter (3 G 4) (Do not wash it before and after filtration), put it into the brown bottle washed with steam for 30 min and store it in a dark place. Determine the factor of aqueous solution at every use.

To determine the factor of this solution, take 100 ml of purified water into an Erlenmeyer flask (300 ml) and accurately add 5 ml of dilute sulfuric acid, several glass beads and 5 ml of potassium permanganate above-mentioned thereto. Then, after boiling for 5 min, extinguish the fire and immediately accurately add 10 ml of 0.01 N sodium oxalate solution and decolourize it.

Thereafter, drop potassium permanganate solution until faint red colour does not disappear and remains in the liquid phase. Then, further accurately add 5 ml of dilute sulfuric acid and 5 ml of potassium permanganate to this solution and boil it for 5 min. Thereafter, add 10 ml of 0.01 N sodium oxalate solution in the same way as that at previous time and immediately titrate it by using potassium permanganate solution until faint red colour does not disappear and slightly remains. Obtain total amount (a , ml in unit) of 5 ml of potassium permanganate solution added at first in the second titration and the amount of potassium permanganate solution required for titration and calculate the factor from the following formula:

$$\text{Factor (F)} = \frac{10}{a}$$

- (4) Glass Beads After preliminarily boiling good quality glass beads of about 2 mm in diameter in sulfuric acid (4 N) for about 30 min, sufficiently wash them with hot purified water until the washings become neutral (affirm it with a litmus paper) and dry them in an oven at 110°C. Thereafter, store them in a glass bottle.
- (5) Clean Erlenmeyer Flask (300 ml) After sufficiently washing, add 100 ml of purified water, 5 ml of dilute sulfuric acid, 10 ml of 0.01 N potassium permanganate solution and several glass beads and boil them for about 5 min. Immediately add 10 ml of 0.01 N sodium oxalate solution and decolourize them. Thereafter, drip 0.01 N potassium permanganate solution until faint red colour does not disappear and slightly remains. Thereafter, carefully discard the solution so that glass beads remain in the flask and immediately use it for test.

4.2 Test Operation Take 100 ml of sample water into a clean Erlenmeyer flask, add 5 ml of dilute sulfuric acid thereto, then add 10 ml of 0.01 N potassium permanganate solution by using a brown bottle and boil it on a wire gauze with asbestos (or on an electric heater) for 5 min (time required until it is boiled, is 5 to 7 min).

Then, extinguish the fire, immediately add 10 ml of 0.01 N sodium oxalate solution from another burette to decolourize it, immediately drip 0.01 N of potassium permanganate solution to this solution and titrate it until faint red colour does not disappear and slightly remains. Thus, obtain total amount (a , ml in unit) of 0.01 N potassium permanganate solution required before and after.

Separately, operate the test regarding 100 ml blank test water under the same conditions as those for sample water, obtain total amount (b , ml in unit) of 0.01 N potassium permanganate solution required for blank test water and calculate the consumption (ppm) of potassium permanganate from the following formula:

$$\text{Consumption of potassium permanganate (ppm)} = (a - b)F \times \frac{1000}{L} \times 0.316$$

where a : 0.01 N potassium permanganate solution required for 100 ml sample water (ml)
 b : 0.01 N potassium permanganate solution required for 100 ml blank test water (ml)

F : factor of 0.01 N potassium permanganate solution

L : sample water and blank test water (ml)

5. Loss of Residual Chlorine

5.1 Reagents and Device The reagents and device shall be as follows:

- (1) Orthotolidine Solution Take 1.35 g of orthotolidine dihydrochloride $[(CH_3 \cdot C_6H_3 \cdot NH_2)_2 \cdot 2HCl]$, dissolve it with about 800 ml of purified water, add 150 ml of hydrochloric acid (35 %), dilute it to 1 l using purified water and store it in a brown bottle.
- (2) Carbon-Dioxide-Free Purified Water It is in accordance with 6. (1.14).
- (3) Buffer Solution Dissolve 22.86 g of disodium phosphate (Na_2HPO_4) and 46.14 g potassium phosphate (KH_2PO_4) preliminarily dried at 105 to 110°C and left cool in a desiccator, with carbon-dioxide-free purified water, dilute it to 1 l, stand it still for several days, filter the deposited precipitate and allow that to be the stock solution.

Then, dilute 400 ml of stock solution to 2 l by adding carbon-dioxide-free purified water and allow that to be the buffer solution. pH of this buffer solution is 6.45.

- (4) Solution of Chromic Acid and Potassium Dichromate Take 4.65 g of potassium chromate (K_2CrO_4) and 1.55 g of potassium dichromate ($K_2Cr_2O_7$) into a measuring flask (1 l), dissolve them with buffer solution and dilute its total amount to 1 l. Stopper this solution and store it in a dark place. When precipitates are generated, filter them with glass filter (G 3) or filter paper (Class 5A). That elapsed 6 months min. shall not be used.
- (5) Residual Chlorine Standard Colorimetric Solution Mix potassium chromate and potassium dichromate solutions at a proportion given in the following Appendix Table, respectively take them into colour comparison tubes of 100 ml and record the concentration (ppm) of corresponding residual chlorine.

Appendix Table. Residual Chlorine Colourimetric Standard Solution
(20 cm in Solution Layer)

Residual chlorine ppm	Solution of potassium chromate and potassium dichromate ml	Buffer solution ml
0.1	1.0	99.0
0.2	2.0	98.0
0.3	3.0	97.0
0.4	4.0	96.0
0.5	5.0	95.0
0.6	6.0	94.0
0.7	7.0	93.0
0.8	8.0	92.0
0.9	9.0	91.0
1.0	10.0	90.0
1.2	12.0	88.0
1.3	13.0	87.0
1.5	15.0	85.0
2.0	19.7	80.3

- (6) Colour Comparison Tube (100 ml) It is the flat bottom non-colour test tube with ground stopper of about 25 cm in total length, marked with the marked line of 100 ml at a height of 20 ± 0.3 cm from the bottom.

5.2 Test Operation Take 5 ml of orthotolidine solution into a colour comparison tube (100 ml), add sample water thereto up to the marked line (1.3 max. in pH) and stand it still for 5 min. Then, compare the coloration of sample water with that of residual chlorine colorimetric standard solution prepared in a colour comparison tube of the same type and obtain the residual chlorine (ppm) of sample water from a corresponding colorimetric standard solution.

Then, obtain the residual chlorine (ppm) of blank test water in the same way and allow the difference of residual chlorine (ppm) between this and that of sample water to be the loss of residual chlorine.

3. Phenols

Because phenols in sample are apt to be decomposed, they shall be tested within 4 h().

Note (1) When the test cannot be commenced within 4 h after sampling, add phosphoric acid solution just after sampling, make pH 4 max., add copper sulfate solution at a proportion of 0.1 % and carry out the test within 24 h.

(1) Reagents and Devices

(1.1) Active Carbon It shall be powdery.

(1.2) Purified Water It shall not contain phenols and residual chlorine. It is prepared by a method wherein purified water is added with active carbon at a rate of 10 to 20 ppm, sufficiently mixed with shaking and stood still for one night and filtered.

For the present tests, this purified water shall be always used.

(1.3) Copper Sulfate Solution Dissolve 10 g of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) with purified water and dilute it to 100 ml.

(1.4) 4-Aminoantipyrine Solution Dissolve 0.25 g of 4-aminoantipyrine [$\text{CH}_3 \cdot \text{C} : \text{CH}(\text{NH}_2)\text{OC} \cdot \text{N}(\text{C}_6\text{H}_5) : \text{N}(\text{CH}_3)$] in purified water, dilute it to 100 ml and store it in cold dark place.

Allow the available period of this solution to be about one week.

(1.5) Potassium Ferricyanide Solution Dissolve 0.5 g of potassium ferricyanide [$\text{K}_3\text{Fe}(\text{CN})_6$] in purified water and dilute it to 100 ml.

This solution shall be prepared at every use.

(1.6) Methyl Orange Indicator Dissolve 0.5 g methyl orange ($\text{C}_{14}\text{H}_{14}\text{N}_3\text{O}_3\text{SNa}$) in purified water to make its total amount 1 l and store it in a brown bottle.

(1.7) Phosphoric Acid Solution Dilute phosphoric acid 10 times with purified water.

(1.8) Phosphoric Acid Buffer Solution Dissolve 104.5 g dipotassium phosphate (K_2HPO_4) and 72.3 g potassium phosphate (KH_2PO_4) in purified water and dilute it to 1 l.

(1.9) Phenol Standard Stock Solution Dissolve 1 g of phenol ($\text{C}_6\text{H}_5\text{OH}$) in purified water, dilute it to 1 l, allow it to be the stock solution and store it in cold dark place by putting it into a brown bottle.

Prepare the standard stock solution from this stock solution at every test.

To prepare standard stock solution, take 50 ml of purified water into an Erlenmeyer flask (300 ml) with ground stopper, accurately add 25 ml of phenol stock solution thereto and mix them. Then, accurately add 25 ml of 0.1 N solution of bromic acid and potassium bromide, stopper it and sufficiently mix it with shaking.

Then, add 3 ml of hydrochloric acid (35 %), stopper it again, sufficiently mix it with shaking and stand it still. After 15 min, add 2 g of potassium iodide (KI). Thereafter, stopper it and sufficiently mix it with shaking for 1 min.

Titrate the deposited iodine herein with 0.1 N sodium thiosulfate solution using starch solution as indicator and obtain the amount (*a*, ml in unit) of sodium thiosulfate solution required therefore. Separately, take 75 ml of purified water into an Erlenmeyer flask, add 25 ml of 0.1 N solution of bromic acid and potassium bromide, 3 ml of hydrochloric acid and 2 g of potassium iodide, titrate the deposited iodine with 0.1 N sodium thiosulfate solution, obtain the amount (*b*, ml in unit) of sodium thiosulfate required therefore and obtain the amount (mg) of phenol in 1 ml stock solution from the following formula:

$$X = \frac{(b-a)F \times 1.569}{25}$$

where X : phenol (ppm)

a : 0.1 N sodium thiosulfate solution required for 25 ml of phenol stock solution (ml)

b : 0.1 N sodium thiosulphate solution required for 25 ml of 0.1 N solution of bromic acid and potassium bromide (ml)

F : factor of 0.1 N sodium thiosulfate solution

Then, accurately take 10/ X ml phenol stock solution into a brown measuring flask of 1 l, dilute it to 1 l by adding purified water and sufficiently mix it. Thereafter, take 100 ml thereof into another measuring flask (1 l) to make its total amount 1 l by adding purified water and allow that to be the standard stock solution.

- (1.10) Starch Solution Sufficiently mix 1 g of starch (potato starch) with 100 ml of purified water, slowly add that into 200 ml hot purified water continuously mixing them with stirring and boil them until the solution becomes semitransparent. Thereafter, stand the solution still and use the supernatant.
- (1.11) 0.1 N Solution of Bromic Acid and Potassium Bromide Take 2.783 g of potassium bromate (KBrO₃) and 20 g of potassium bromide (KBr) preliminarily dried at 100°C and left cool in sulfuric acid desiccator into a measuring flask (1 l), dissolve them in purified water and make its total amount 1 l.
- (1.12) 0.1 N Solution of Thiosulfate Take 26 g of sodium thiosulfate (Na₂S₂O₃ · 5H₂O) and 0.2 g of sodium carbonate (Na₂CO₃) and dissolve them in about 1 l of carbon-dioxide-free purified water. Thereafter, add 10 ml of isoamyl alcohol [(CH₃)₂CH CH₂CH₂OH] to make its total amount 1 l. After sufficiently mixing it with shaking, stopper it and stand it still for 2 days. Thereafter, determine its factor.

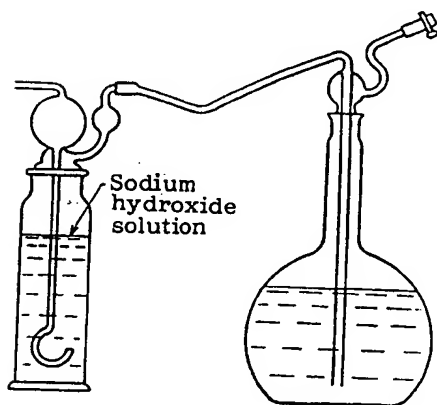
To determine the factor of this solution, accurately take 25 ml of 0.1 N potassium iodate solution into an Erlenmeyer flask (300 ml) with ground stopper, add 2 g of potassium iodide (KI) and 5 ml of sulfuric acid (6 N), immediately stopper it, mix it with quietly shaking and stand it still in a dark place for 5 min. Thereafter, add about 100 ml of purified water and titrate the liberated iodine using above-mentioned sodium thiosulfate solution. When brown colour turns light yellow colour, add starch solution and continue to titrate it until the generated blue colour disappears. Obtain the amount (a , ml in unit) of sodium thiosulfate solution required therefore and calculate its factor from the following formula:

$$\text{Factor } (F) = \frac{25}{a}$$

Remark: The consumption of sodium thiosulfate solution shall be calibrated by separately carrying out blank test.

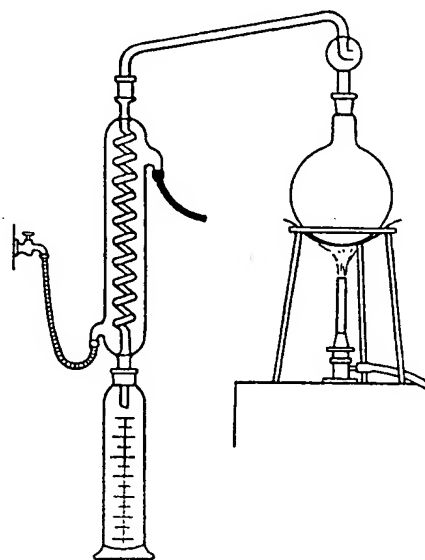
- (1.13) 0.1 N Potassium Iodate Solution Take 3.567 g of standard reagent potassium iodate (KIO_3) which is preliminarily dried at 120 to 140°C for 1.5 to 2 h and thereafter stood to cool in a sulfuric acid desiccator, into a measuring flask and make its total amount 1 l.

Appendix Fig. 1



Carbon-dioxide-free Purified Water
Storage Equipment

Appendix Fig. 2



Distilling Equipment

- (1.14) Carbon-Dioxide-Free Purified Water After removing carbon dioxide gas and other volatile matters by boiling the purified water redistilled, leave the purified water to cool to ordinary temperature not so as to absorb carbon dioxide gas from the air. Store the purified water as shown in Appendix Fig. 1.

(1.15) Distilling Equipment It is as given in Appendix Fig. 2. It is made of glass, its ground joint being interchangeable joint and the inner capacity of distillation flask is 300 to 500 ml.

(1.16) Glass Beads Those are in accordance with 4.1 (4).

(2) Test Operation Take 200 ml of test water (the amount containing 0.2 to 20 μg as phenol) into a distilling flask, add copper phosphate solution⁽²⁾, several drops of methyl orange indicator and several glass beads thereto and add phosphoric acid solution until the colour of solution turns red. Thereafter, distil it.

When the remainder solution becomes about 180 ml, stop distillation, add 20 ml of purified water thereto after the liquid in the distilling flask does not boil, continue to distil it again, further distil 20 ml and allow total remainder to be 200 ml.

Add 10 ml of phosphoric acid buffer solution thereto and blend them. Thereafter, make pH 9.5 (± 0.2)⁽³⁾ by adding aqueous ammonia (10 N) and transfer it to a separating funnel (300 ml). Thereafter, add 1 ml of 4-aminoantipyrin, sufficiently blend them, then add 2.5 ml of potassium ferricyanide solution, sufficiently blend them and stand it still for 10 min.

Then, add 25 ml of chloroform and mix them by strongly shaking for 30 sec. Thereafter, stand it still for 5 min, separately take the layer of chloroform, filter⁽⁴⁾ it with a dry filter paper (Class 3) and allow that to be the inspection solution.

Separately, take 200 ml of blank test water into a beaker and separately take a suitable amount (0.1 to 20 ml) of phenol standard stock solution into several beakers. Respectively add purified water, make the total amount 200 ml, hereinafter treat it in the same way as that for the inspection solution and allow those to be the blank test solution and the standard solution.

Then, take inspection solution blank test solution and standard solution into absorption cells (40 mm min.), measure the absorbancy at 460 μm wavelength according to absorptiometry and obtain phenols in sample water as phenol (ppm) according to the working curve from the absorbancies of inspection solution and blank test solution obtained herein.

Notes ⁽²⁾ For the stock sample preliminarily added with copper sulfate solution, this operation is omitted.

⁽³⁾ The use of pH meter is convenient.

⁽⁴⁾ Before taking separately the layer of chloroform separated, wipe the water content at the leg part of separating funnel shall be wiped off with finely crumpled filter paper.

7. Amine

7.1 Reagents The reagents shall be as follows:

(1) *p*-dimethylaminobenzaldehyde $(\text{CH}_3)_2\text{NC}_6\text{H}_4\text{CHO}$, powder

(C) Toluene $\text{CH}_3\text{C}_6\text{H}_5$

7.2 Test Operation Take 1 ml of specimen water into a test tube, add 10 to 20 mg of *p*-dimethylaminobenzaldehyde thereto, then add 0.5 ml of toluene and blend them. Thereafter, add 0.1 ml of sulfuric acid (95 %), stand it still for 1 min, further add 1 ml of ethyl alcohol and allow that to be the inspection solution. Operate blank test water in the same way as that for specimen water and allow it to be the blank test solution.

Then, compare the colour of inspection solution with the colour of blank test solution. When amine exists at that time, yellow, purple or pink colour thicker than the coloration of blank test solution appears.

8. Cyan

8.1 Reagents The reagents shall be as follows:

(1) Buffer Solution Dissolve 3.40 g of potassium phosphate (KH_2PO_4) and 3.55 g of disodium phosphate (anhydrous) (Na_2HPO_4) in purified water and dilute it 1 l.

(2) Chloramine T Solution Dissolve 1.25 g of chloramine T ($\text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{NCINa}\cdot 3\text{H}_2\text{O}$) with purified water and dilute it to 100 ml.

Prepare this solution at every use.

(3) Mixed Solution of Pyridine and Pyrazolone Dissolve 0.5 g of 1-phenyl-3-methyl-5-pyrazolone ($\text{C}_{10}\text{H}_{10}\text{ON}_2$) in 200 ml purified water at about 70°C and cool it to room temperature (in that case, it may be not completely dissolved). Separately, dissolve 0.04 g bis-(1-phenyl-3-methyl-5-pyrazolone) ($\text{C}_{20}\text{H}_{18}\text{O}_2\text{N}_4$) in 40 ml of pyridine ($\text{C}_5\text{H}_5\text{N}$), add it to that solution and blend them.

Prepare this solution at every use.

8.2 Test Operation Take 20 ml of inspection water (the amount containing 0.0002 to 0.01 mg as (CN) into a colour comparison tube (50 ml). Then, add 10 ml of buffer solution and 0.25 ml of chloramine T solution to the inspection water, immediately stopper it, mix it by quietly shaking and leave it as it is for 2 to 3 min. Thereafter, add 15 ml of the mixed solution of pyridine and pyrazolone, sufficiently blend them and stand it still at 20 to 30°C for 30 min. When cyan exists, steady blue appears via purple from light red.

The sensitivity of this method is about 0.01 ppm.

Remarks 1. The cyans measured by this method are derived from cyan ion, hydrogen cyanide, cyan chloride or the like.

2. In this method, the sample is coloured also by thiocyanate ion (SCN^-).

9. Odour and Taste

9.1 Test Operation Respectively take about 100 ml of sample water and blank test water into Erlenmeyer flasks with ground stopper, lightly stopper them, heat them at 40 to 50 °C and inspect the presence of odour and taste by comparing the sample with reference blank test water just after opening the stopper.

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Method of Erichsen cupping test

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS Z 2247 : 1993** is replaced with **JIS Z 2247 : 1998**.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Method of Erichsen cupping test

Introduction This Standard is the Japanese Industrial Standard prepared based on ISO 8490-1986, Metallic materials — Sheet and strip — Modified Erichsen cupping test, in such a manner that the corresponding parts in the corresponding International Standard are contained without any modification in the technical content, and the item (Erichsen test method A) not specified in the corresponding International Standard is complementarily provided as a part of the Japanese Industrial Standard. However, this part is to be withdrawn by the year 2003.

1 Scope This Japanese Industrial Standard specifies the method of determining the Erichsen value of metallic sheets having a thickness of 0.1 mm to 2.0 mm and a width of 90 mm or more.

Remarks : The International Standard corresponding to this Standard is as follows :

ISO 8490 : 1986 *Metallic materials — Sheet and strip — Modified Erichsen cupping test*

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute a part of the provisions of this Standard. The most recent editions of the standards indicated shall be applied.

JIS B 7729 *Erichsen cupping testers*

JIS G 0202 *Glossary of terms used in iron and steel (testing)*

JIS Z 8401 *Rules for rounding off of numerical values*

3 Definitions For the purposes of this Standard, the definitions given in JIS G 0202 and the following definitions apply.

- a) **Erichsen test method** The Erichsen test method to be performed by clamping a test piece between a die and a blank holder with a constant holding force of approximately 10 kN.
- b) **Erichsen value** The value expressed in millimeters for the moved distance of the end of the punch from the surface of the blank holder until a through crack reaching the back surface of the test piece appears at least at a part of the test piece during the course of the Erichsen test.

Remarks : Erichsen test method A This is the Erichsen test method to be performed with a clearance of 0.05 mm between a die and a blank holder besides a test piece thickness. This test method may be applicable for steel by the year 2003, and its designation shall be the symbol A prefixed to the Erichsen value.

Example : A10.2

4 Principle Form a cup shape by pressing a punch with a spherical end against a clamped test piece between a blank holder and a die, and express in millimeters the moved distance of the end of the punch from the surface of the blank holder until a through crack reaching the back surface of the test piece appears at least at a part of the test piece.

5 Test pieces The test pieces shall be classified into three types of Nos. 1 to 3 by shape, and the dimensions shall be as given in Table 1. The types of the test pieces used shall be clearly stated in the test results.

Table 1 Dimensions of test pieces

Unit : mm

Type	Dimensions
No. 1 test piece	Strip (band) of 90 ± 2 in width
No. 2 test piece	Square of 90 ± 2 in the length of side
No. 3 test piece	Circle of 90 ± 2 in diameter

6 Testing machine The testing machine shall be as specified in JIS B 7729

7 Erichsen test The Erichsen test shall be performed as follows :

- The test piece shall be greased and the graphite grease shall ordinarily be used.
- The test piece shall be so placed that the centre line of the width or the centre of the square or the circle is in accord with the centre of the punch or die. In the case of strip (band), the distance between the centres of dents shall be 90 mm or over.
- The indication of the micrometer shall be set to zero at the position where the end of the punch stands on the same plane with the surface of the blank holder, or the difference shall be used for purpose of correction.
- The pressing speed of the punch shall generally be at a rate of 5 mm/min to 20 mm/min, being uniform at first, then gradually decreased towards the end of the test and eventually to the minimum of the rate in the vicinity where a crack is likely to appear.
- Judgment of the appearance of a crack shall be made by the visual, mechanical or electrical method. However, in the case of the mechanical or electrical method, correspondence with the visual confirmation of crack appearance shall sufficiently be performed.
- The testing temperature shall be within the limits 10°C to 35°C , and if necessary, shall be recorded. However, the test carried out under controlled conditions shall be made at a temperature of $(23\pm 5)^{\circ}\text{C}$ as standard, and, for materials sensitive to temperature changes, the requirements of the material's JIS standards shall be applied.

8 Expression of Erichsen value The Erichsen value shall be rounded off to one decimal place in accordance with JIS Z 8401.

9 Report of test results When the test report is necessary, selection shall be made among the following items by agreement between the purchaser and manufacturer.

- Reference to this Standard
- Identification of the test piece
- Thickness of the test piece
- Type of lubricant used
- Erichsen value
- Testing temperature

Informative reference : The graphite grease to be used for the Erichsen test shall be composed of calcium soap, refined mineral oil and graphite, and its properties are preferably as shown in Informative reference Table 1. It shall be free from corrosive matters, granular resin and other foreign matters.

Informative reference Table 1 Graphite grease

Items	Properties	
Grease	Cone penetration	250 to 280 (depth per 0.1 mm)
	Free acid	Oleic acid 0.2 % (m/m) max.
	Free alkali	Ca(OH) ₂ 0.3 % max.
	Water content	0.5 % to 1.2 % (m/m)
	Graphite content	23 % to 28 %
Graphite flake	Average particle size	0.3 mm max.
	Maximum particle size	0.5 mm max.
	Ash	4.5 % (m/m) max.
Mineral oil	Kinematic viscosity at 37.8°C	100 cSt to 120 cSt
	Flash point	177 °C min.
	Ash	0.01 % (m/m) max.
	Neutralization value	0.1 mgKOH/g max.

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